

Appendix A Pending Claim Set Serial No.: 09/014,341 Filed: 27 January 1998

For: ANTISOILING COATINGS FOR ANTIREFLECTIVE SURFACES AND METHODS OF PREPARTION

1. An antireflective article comprising a substrate having an antireflective surface and an antisoiling coating that is at least partially cured thereon; wherein the antisoiling coating comprises a fluorinated siloxane prepared by applying a coating composition comprising at least one fluorinated silane having a number average molecular weight of at least about 1000 and the following formula:

$$R_{f}$$
-[- R^{1} -SiY_{3-x} R^{2}_{x}]_y
(I)

wherein: R_f is a monovalent or divalent polyfluoropolyether group; R^1 is a substituted divalent alkylene group, arylene group, or combinations thereof, wherein at least one substituent in R^1 is selected from the group consisting of heteroatoms and functional groups, and further wherein at least one substituent in R^1 is optionally a halogen; R^2 is a C_1 - C_4 alkyl group; Y is a halogen, a C_1 - C_4 alkoxy group, or a C_1 - C_4 acyloxy group; x is 0 or 1; and y is 1 or 2.

- 2. The antireflective article of claim 1 wherein the antireflective surface comprises a metal oxide film having one or more metal oxides.
- 3. The antireflective article of claim 2 wherein the antireflective surface comprises a vacuum deposited metal oxide film.
- 4. The antireflective article of claim 3 wherein the antisoiling coating is at least about 15 Angstroms thick.
- 5. The antireflective article of claim 4 wherein the antisoiling coating is no greater than about 150 Angstroms thick.

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6. The antireflective article of claim 1 which has a first surface antireflectivity that is different by less than about 0.5 percentage units from that of the same article without the antisoiling coating.

- 7. The antireflective article of claim 1 wherein the coating is prepared by applying a coating composition comprising at least one fluorinated silane wherein each Y is a C₁-C₄ alkoxy group.
- 8. The antireflective article of claim 1 wherein the coating is prepared by applying a coating composition comprising at least one fluorinated silane wherein R_f is a perfluoropolyether group.
- 9. The antireflective article of claim 8 wherein the coating is prepared by applying a coating composition comprising at least one fluorinated silane wherein R_f is a perfluoropolyether group comprising perfluorinated repeating units selected from the group consisting of $-(C_nF_{2n})$, $-(C_nF_{2n}O)$, -(CF(Z)), -(CF(Z)O), $-(CF(Z)C_nF_{2n}O)$, $-(C_nF_{2n}CF(Z)O)$, and combinations thereof, wherein n is 1 to 4 and Z is a perfluoroalkyl group, a perfluoroetheralkyl group, a perfluoroalkoxy group, or a perfluoroetheralkoxy group, each of which has about 1 to about 9 carbon atoms and 0 to about 4 oxygen atoms.
- 10. The antireflective article of claim 1 wherein the coating is prepared by applying a coating composition comprising a fluorinated silane selected from the group consisting of XCF₂O(CF₂O)_m(C₂F₄O)_pCF₂X, C₃F₇O(CF(CF₃)CF₂O)_pCF(CF₃)X, XCF(CF₃)O(CF(CF₃)CF₂O)_pCF(CF₃)X, XCF₂O(C₂F₄O)_pCF₂X, CF₃O(C₂F₄O)_pCF₂X, X(CF₂)₃O(C₄F₈O)_p(CF₂)₃X, and mixtures thereof, wherein:

-X is $-R^1$ -SiY_{3-x} R^2 _x as defined in claim 1 or a terminal group selected from the group consisting of (C_nF_{2n+1}) -, $(C_nF_{2n+1}O)$ -, $(X'C_nF_{2n}O)$ -, and $(X'C_nF_{2n})$ -, wherein X' is H, Cl, or Br, with the proviso that at least one X group per molecule is a silane, and n is 1 or more;



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an average value of m is within a range of about 1 to about 50; and an average value of p is within a range of about 4 to about 40.

- 11. The antireflective article of claim 1 wherein the antisoiling coating composition further includes an alkyl perfluoroalkyl ether.
- 12. The antireflective article of claim 1 wherein R¹ includes about 2 to about 16 carbon atoms.
- 13. An antisoiling coating composition comprising at least one fluorinated silane having a number average molecular weight of at least about 1000 and an alkyl perfluoroalkyl ether, wherein the fluorinated silane has the following formula:

$$R_{\mathcal{F}}[-R^1-SiY_{3-x}R^2_x]_y$$
(I)

wherein: R_f is a monovalent or divalent polyfluoropolyether group; R^1 is a substituted or unsubstituted divalent alkylene group, arylene group, or combinations thereof, wherein at least one substituent if present is selected from the group consisting of heteroatoms, functional groups, and halogens; R^2 is a C_1 - C_4 alkyl group; Y is a halogen, a C_1 - C_4 alkoxy group, or a C_1 - C_4 acyloxy group; X is 0 or 1; and X is 1 or 2.

14. The antisoiling composition of claim 13 wherein R_f has an approximate average structure selected from the group consisting of $-CF_2O(CF_2O)_m(C_2F_4O)_pCF_2$ -, $C_3F_7O(CF(CF_3)CF_2O)_pCF(CF_3)$ -, $CF_3O(C_2F_4O)_pCF_2$ -, - $CF(CF_3)O(CF(CF_3)CF_2O)_pCF(CF_3)$ -,

-CF₂O(C_2F_4O)_pCF₂-, and -(CF₂)₃O(C_4F_8O)_p(CF₂)₃-, wherein m has an average value of 0 to about 50, and p has an average value of 0 to about 50, with the proviso that both m and p cannot be 0 in the same group.



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15. A method of applying an antisoiling coating to a substrate having an antireflective surface, the method comprising treating the antireflective surface with a coating composition comprising at least one fluorinated silane having a number average molecular weight of at least about 1000 and the following formula:

$$R_{\mathcal{F}}[-R^1-SiY_{3-x}R^2_x]_y$$
(I)

wherein: R_f is a monovalent or divalent polyfluoropolyether group; R^1 is a substituted divalent alkylene group, arylene group, or combinations thereof, wherein at least one substituent in R^1 is selected from the group consisting of heteroatoms and functional groups, and further wherein at least one substituent in R^1 is optionally a halogen; R^2 is a C_1 - C_4 alkyl group; Y is a halogen, a C_1 - C_4 alkoxy group, or a C_1 - C_4 acyloxy group; x is 0 or 1; and y is 1 or 2.

- 16. The method of claim 15 wherein the coating composition further comprises a nonchlorinated solvent selected from the group consisting of a fluorinated alkane, an alkyl perfluoroalkyl ether, and mixtures thereof.
- 17. The method of claim 15 wherein the coating is prepared by applying a coating composition comprising a fluorinated silane selected from the group consisting of $XCF_2O(CF_2O)_m(C_2F_4O)_pCF_2X$, $C_3F_7O(CF(CF_3)CF_2O)_pCF(CF_3)X$, $XCF(CF_3)O(CF(CF_3)CF_2O)_pCF(CF_3)X$, $XCF_2O(C_2F_4O)_pCF_2X$, X

-X is $-R^1$ -SiY_{3-x} R^2 _x as defined above in claim 15 or a terminal group selected from the group consisting of (C_nF_{2n+1}) -, $(C_nF_{2n+1}O)$ -, $(X'C_nF_{2n})$ -, and $(X'C_nF_{2n})$ -, wherein X' is H, Cl, or Br, with the proviso that at least one X group per molecule is a silane, and n is 1 or more;

an average value of m is within a range of about 1 to about 50; and an average value of p is within a range of about 4 to about 40.



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18. The method of claim 15 wherein the antisoiling coating formed is at least about 15 Angstroms thick.

- 19. The method of claim 18 wherein the antisoiling coating formed is no greater than about 150 Angstroms thick.
- 20. The method of claim 15 wherein the step of treating comprises coating the composition at room temperature followed by heating the coated composition at a temperature of at least about 100°C.
- 21. The method of claim 15 wherein the coating composition comprising the fluorinated silane comprises less than about 2.0 weight percent of the fluorinated silane.
- 22. The method of claim 15 wherein the step of treating comprises continuously roll coating the composition onto the substrate.
- 23. (Amended) The method of claim 22 wherein the step of [gravure] <u>roll</u> coating comprises feeding the coating composition to a doctor blade, transferring the coating composition from the doctor blade to a gravure roll, and applying the coating composition to the antireflective surface of the substrate from the gravure roll.
- 24. The method of claim 23 wherein the step of coating the antisoiling coating composition further comprises applying a soft roll to a surface opposing the antireflective surface of the transparent substrate.
- 25. The method of claim 22 wherein the antisoiling coating composition further comprises a nonchlorinated solvent.



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26. The method of claim 25 wherein the solvent is selected from the group consisting of a fluorinated alkane, an alkyl perfluoroalkyl ether, and mixtures thereof.

- 27. The method of claim 26 wherein the solvent is an alkyl perfluoroalkyl ether.
- 28. An antireflective article made by the method of claim 22.
- 29. An antireflective article comprising:

a transparent substrate having a first surface and a second surface; an antireflective coating on at least a portion of the first surface; and an antisoiling coating disposed on the antireflective coating, wherein the antisoiling coating comprises siloxane groups and polyfluoropolyether segments covalently bonded to silicon via organic linking groups, wherein the polyfluoropolyether segments have a molecular weight of at least about 1000 and the organic linking groups include nitrogen atoms.

- 31. The antireflective article of claim 29 wherein the antisoiling coating has a fluorine atom to nitrogen atom ratio of about 25 to about 150.
- 32. The antireflective article of claim 29 wherein the antisoiling coating comprises a fluorinated siloxane prepared by applying a coating composition comprising at least one fluorinated silane having a number average molecular weight of at least about 1000 and the following formula:

$$R_{f}[-R^{1}-SiY_{3-x}R^{2}_{x}]_{y}$$
(I)

wherein: R_f is a monovalent or divalent polyfluoropolyether group; R^1 is a nitrogensubstituted divalent alkylene group, arylene group, or combinations thereof, optionally substituted with one or more substituents selected from the group consisting of heteroatoms other than nitrogen, functional groups, and halogens; R^2 is a C_1 - C_4 alkyl



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group; Y is a halogen, a C_1 - C_4 alkoxy group, or a C_1 - C_4 acyloxy group; x is 0 or 1; and y is 1 or 2.

- 33. The antireflective article of claim 32 wherein the R¹ group contains heteroatoms or functional groups and is optionally substituted with halides.
- 34. The antireflective article of claim 33 wherein R¹ is a divalent hydrocarbon containing at least one functional group.
- 35. The antireflective article of claim 29 wherein the transparent substrate comprises a flexible organic polymeric material.
- 36. The antireflective article of claim 35 further comprising an adhesion-enhancing coating disposed between the flexible organic polymeric substrate and the antireflective coating.
- 37. The antireflective article of claim 36 wherein the antireflective coating comprises a metal oxide film having one or more metal oxides.
- 38. The antireflective article of claim 37 wherein the antireflective surface comprises a vacuum deposited metal oxide film.
- 39. An antireflective article comprising:
- a transparent substrate comprising a flexible organic polymeric material having a first surface and a second surface;

an antireflective coating on at least a portion of the first surface;

a layer of a pressure sensitive adhesive disposed on the second surface of the substrate; and

an antisoiling coating on at least a portion of the antireflective coating, wherein the antisoiling coating comprises siloxane groups and polyfluoropolyether segments



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covalently bonded to silicon via organic linking groups, wherein the polyfluoropolyether segments have a molecular weight of at least about 1000.

40. An antireflective article comprising a substrate having an antireflective surface and an antisoiling coating that is at least partially cured thereon; wherein the antisoiling coating comprises a fluorinated siloxane prepared by applying a coating composition comprising at least one fluorinated silane having a number average molecular weight of at least about 1000 and the following formula:

$$R_{\mathcal{F}}[-R^1-\mathrm{Si}Y_{3-x}R^2_{x}]_y$$
(I)

wherein: R_f is a monovalent or divalent polyfluoropolyether group; R^1 is a substituted or unsubstituted divalent alkylene groups, arylene group, or combinations thereof, wherein at least one substituent if present is selected from the group consisting of heteroatoms, functional groups, and halogens; R^2 is a C_1 - C_4 alkyl group; Y is a halogen, a C_1 - C_4 alkoxy group, or a C_1 - C_4 acyloxy group; X is 0 or 1; and X is 1 or 2 and further wherein the antisoiling coating composition includes an alkyl perfluoroalkyl ether.

41. An antireflective article comprising a substrate having an antireflective surface and an antisoiling coating that is at least partially cured thereon; wherein the antisoiling coating comprises a fluorinated siloxane prepared by applying a coating composition comprising at least one fluorinated silane having a number average molecular weight of at least about 1000 and the following formula:

$$R_{f} = [-R^1 - SiY_{3-x}R^2_x]_y$$
(I)

wherein: R_f is a divalent polyfluoropolyether group; R^1 is a substituted or unsubstituted divalent alkylene group, arylene group, or combinations thereof, wherein at least one substituent if present is selected from the group consisting of heteroatoms, functional

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groups, and halogens; R^2 is a C_1 - C_4 alkyl group; Y is a halogen, a C_1 - C_4 alkoxy group, or a C_1 - C_4 acyloxy group; x is 0 or 1; and y is 2.

42. A method of applying an antisoiling coating to a substrate having an antireflective surface, the method comprising treating the antireflective surface with a coating composition comprising at least one fluorinated silane having a number average molecular weight of at least about 1000 and the following formula:

$$R_{f}[-R^{1}-SiY_{3-x}R^{2}_{x}]_{y}$$
(I)

wherein: R_f is a divalent polyfluoropolyether group; R^1 is a substituted or unsubstituted divalent alkylene group, arylene group, or combinations thereof, wherein at least one substituent if present is selected from the group consisting of heteroatoms, functional groups, and halogens; R^2 is a C_1 - C_4 alkyl group; Y is a halogen, a C_1 - C_4 alkoxy group, or a C_1 - C_4 acyloxy group; X is 0 or 1; and X is 2.

- 45. (New) The antireflective article of claim 1 wherein the antisoiling coating composition further comprises a solvent.
- 46. (New) The method of claim 15 wherein the antisoiling coating composition further comprises a solvent.

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